National Bureau of Standards

TECHNICAL NEWS BULLETIN

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Technology

Disintegration Rate of Sodium 22

In preparing standards of radioactivity and in using radioactive isotopes as biological tracers, it is important to know the number of atoms disintegrating in each unit of time. One way of determining this, for radioactive substances emitting both beta and gamma radiation, is to count the beta and gamma emissions that occur simultaneously. For most radioactive substances this can be done with an experimental arrangement consisting only of a radioactive source, a beta-ray counter, and a gamma-ray counter. But in the case of sodium 22, decaying to neon 22, the measurement problem becomes more difficult because each atom of sodium 22 disintegrates by emitting a positron instead of the usual electron. Positrons are very readily absorbed by matter and converted into a form of gamma radiation known as "annihilation radiation," and the gamma counter cannot distinguish this radiation from the nuclear gamma radiation that accompanies beta emission. In order to overcome this difficulty, J. L. Herson of the National Bureau of Standards has used a coincidence counting arrangement in which the beta and gamma counters are separated from each other by a lead block and placed in a moderate magnetic field. The radioactive source is placed near the gamma counter so that positrons emitted by the source will travel, under the influence of the magnetic field, in a semicircular path around the lead block to the beta counter. The lead block effectively prevents the annihilation radiation produced near the beta counter from reaching the gamma counter and causing false coincidences.

The problem of determining the relative number of nuclear and annihilation gamma rays reaching the gamma counter still remains, as a small amount of annihilation radiation is produced near the radioactive source. This was taken into account in the work on sodium 22 at this Bureau by performing the experiment in four different ways. Gamma counting rates were determined with two different gamma counters, observing the gamma rates for each counter with and without an aluminum cover over the source. The two gamma counters were the same shape and size, differing principally in relative counting efficiency for low-energy radiation. The difference in efficiency was caused by using different outer electrode materials—copper in one counter, platinum in the other. The aluminum shield was placed over the source to increase the amount of annihilation radiation. The aluminum does not, however, appreciably affect the number of nuclear gamma rays reaching the counter.

The results of the four experiments yield the information needed to determine (1) the true counting rate for 1.3 million-electron-volts nuclear gamma rays, (2) the counting rate for 0.5 million-volt annihilation gamma rays, (3) the relative efficiency of the two counters for 1.3 million-volt nuclear gamma rays, and (4) the relative efficiency of the two counters for 0.5 million-volt annihilation gamma rays.

The true nuclear gamma count combines with the beta count and the beta-gamma coincidence rate to make possible the computation of the absolute disinte-

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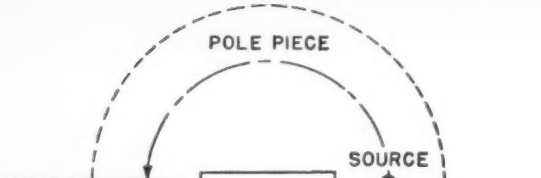


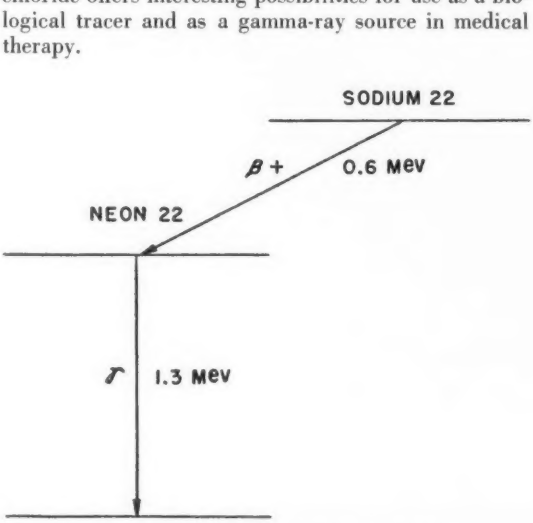
Coincidence counting arrangement.

gration rate of sodium 22. If the number of atoms disintegrating per unit time is designated by N, the number of counts produced in the beta counter will be Np, where p is the probability of a beta ray entering the counter and being counted. Similarly, the true number of nuclear gamma counts is represented by Nq, where q is the corrected probability obtained by using two gamma counters, each with and without an aluminum screen over the source. The probability of both the beta and gamma counters responding to radiation resulting from the disintegration of a single atom is then the product of the respective probabilities, so that the number of genuine coincidences per unit time is Npq. The counting rates, Np, Nq, and Npq, then give three equations in three unknowns, readily solved for the disintegration rate N as well as the probabilities p and q. The disintegration rate for the radioactive sodium chloride solution used in these experiments at this Bureau turned out to be 2.69×10^4 disintegrations per second per milliliter.

Relative Efficiencies

The efficiency of the platinum counter used in this work was found to be practically equal to that of the copper counter for 1.3 million-volt gamma rays, but 2.9 times as large for 0.5 million-volt annihilation radiation. When the source was covered with $\frac{1}{32}$-inch aluminum, the counting rate for annihilation quanta increased by 25 percent. When the copper-electrode counter was used to measure the radiation from sodium 22, about 70 percent of the total gamma counts were due to nuclear gamma rays and 30 percent to annihilation radiation.

The results obtained in this work on sodium 22 at this Bureau agree to within a fraction of 1 percent with the results of an independent method using electrometer measurements against a known radioactive standard. The Bureau's new method for the calibration of positron emitters promises to be quite useful, especially since sodium 22 in the form of sodium chloride offers interesting possibilities for use as a biological tracer and as a gamma-ray source in medical therapy.



Disintegration scheme of sodium 22.

TECHNICAL NEWS BULLETIN

U. S. Department of Commerce
Charles Sawyer, *Secretary*
National Bureau of Standards
E. U. Condon, *Director*

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Stability Test for Additive-Motor Oils

A new evaluation test for lubricating oils with additives, which simulates the characteristic deposits found in engine tests of motor oils, has been developed in the Bureau's lubrication laboratory by S. A. McKee and Ann Robinson Fritz. The method not only offers a sensitive laboratory measure of the effectiveness of various additives in motor oils but also has several advantages over the present testing procedure, which involves disassembly of the test engine and an examination of its parts.[1] The new test, requiring but simple laboratory apparatus, is time saving, is less expensive, and eliminates the fuel variable that results from contamination of the oil by engine fuel in the conventional method. Most important, it gives a quantitative as well as a visual rating of an oil, providing more consistent results and reducing the subjectivity of individual operators.

Such a stability test for additive-treated materials may prove useful as a quick check for control purposes. Many companies now prohibited by cost from making engine tests may also find it an inexpensive method for checking their oil purchases.

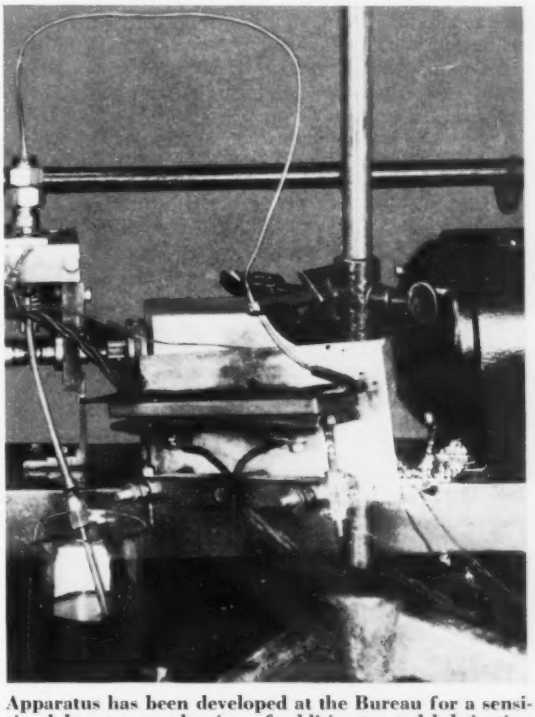
The new test method consists essentially of circulating the oil sample in a thin film over a heated steel strip, quite similar to the oil film on an engine cylinder wall. A mechanical lubricator pump delivers the oil from a small glass container to the upper end of the thin steel strip, which is held in an inclined position and maintained at the test temperature. The oil leaving the lower end of the plate drops into the reservoir and is recirculated. The amount of deposit on this strip provides a sensitive means for rating the lubricating oil under test.

The steel strip is heated by a 7-inch, 200-watt electric heater supported in such a manner that the upper surface of the heater may be inclined at any desired angle toward the reservoir of test oil. The test specimen, 9 inches long, 1¾ inches wide, and 1/32 inch thick, is fastened to the top of a copper bar, which is clamped to the heater. A steel overstrip 3 inches long, 1¾ inches wide, and 1/32 inch thick is fastened at the upper end. overlapping the test strip by 2½ inches. A piece of doubled 20-mesh steel wire screen is placed on the overstrip to assist in spreading the oil evenly on the test strip.

Tests are usually carried out with two units simultaneously. The circulating pump for each unit is operated by a common camshaft driven by a 1/8-horsepower electric motor through a 100 to 1 gear speed reducer. The camshaft, turning at 18 revolutions per minute, has 2 cams for each positive displacement pump, which therefore operates at 36 displacements a minute. The suction line of the pump is fitted with a fine wire-mesh strainer, which is immersed in the reservoir of test oil, and the discharge is through 1/8-inch copper tubing. This may be adjusted to conduct the oil to the overstrip during a test run or to one side for cleaning or calibrating operations. A special head of ¼-inch copper tubing, compressed at one end to form a slit 1/32 inch by ½ inch, is fitted over the tubing that delivers oil to the overstrip.

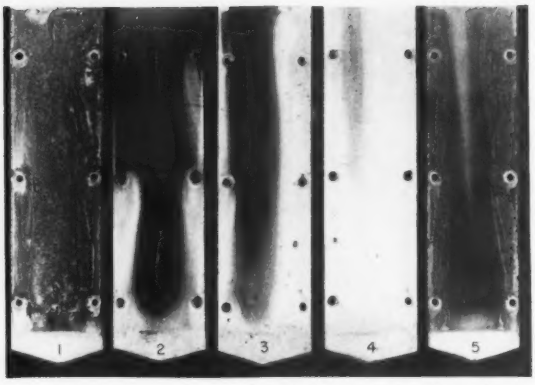
The test temperature, which is measured by a thermocouple inserted in the side of the copper bar at midpoint, is controlled manually by a variable transformer in the line to the heater. The complete assembly is enclosed in a hood with a restricted air inlet so that no forced draft is used, although fumes are removed from the room. This arrangement provides a considerable degree of flexibility. The temperature of the strip may be changed by regulating the transformer. The film of oil flowing down the strip may be varied by changing the slope of the strip and by adjusting the stroke of the pump to change the rate of circulation.

In general, the oils used in the Bureau's investigation are in commercial production and have a considerable background of service performance. They include oils with no additives, premium-type additive oils containing inhibitors and/or detergents, and full heavy-duty oils containing additives that meet U. S. Army Ordnance 2–104B and Navy 9000 series specifications.



Apparatus has been developed at the Bureau for a sensitive laboratory evaluation of additive-treated lubricating oils. This method, which simulates the characteristic engine deposit in engine testing of motor oils, provides results that correlate well with actual engine performance.

[1] For further technical details see Stability test for additive treated motor oils, by S. A. McKee and Ann Fritz, Anal. Chem. 21, 568 (1949).



The effectiveness of various additives in a midcontinent, solvent-extracted oil, is revealed by a new stability test developed at the Bureau: (1) Straight oil (no additive); (2) oxidation inhibitor, no detergent; (3) premium additive with some inhibitor and detergent; (4) heavy-duty additive with full amount inhibitor and detergent; (5) detergent but no inhibitor.

The Bureau's tests were made with weighed samples of 150 milliliters of oil in 250-milliliter beakers under the following conditions: Temperature, 250° C; oil delivery, approximately 0.7 grams per minute; and slope of strip, 8.5 degrees from the horizontal. This temperature approximates the high-temperature regions in the engine under full-power operation. After a heating-up period of about one-half hour, the apparatus is held at the test temperature during a 6-hour run. The test strip is then removed and drained overnight in a vertical position. After cleaning the back of the strip with a naphtha-moistened cloth, the strip is weighed. Following this the copper bar, screens, and steel screen are cleaned, and the strip is again secured in test position for a second 6-hour run. When the test is completed, the strip is removed and allowed to drain at least 24 hours before cleaning the back and weighing.

The difference in weight of the strip before and after the test provides a numerical indication of the amount of deposit formed, but these values are influenced by any oil remaining on the deposit. However, the oil may be removed by a simple method. The test strip is supported in a slightly inclined position over a waste pan and a piece of cotton gauze placed across the strip near the top. A container of precipitation naphtha is located slightly higher than the strip with a wick of gauze from the container feeding naphtha to the gauze on the strip. This provides an even, gentle flow of naphtha down the strip but does not disturb the deposit. When the naphtha dripping from the strip is colorless, the gauzes are removed and the strip dried and weighed. After the test, the oil remaining in the container is weighed, the loss in weight providing an approximate indication of the evaporation loss. The viscosities at 100° F are also measured before and after heating.

Data obtained with the new test give a sensitive indication of the effectiveness of inhibitors, detergents, and their combinations. The particular conditions under which the Bureau tests were conducted tend to accentuate the effectiveness of full heavy-duty additive oils. The apparatus is sufficiently flexible, however, to cover a wide range of conditions that might be more suitable for premium-grade oils, or that would give additional information on the overall effectiveness of a given oil-additive combination.

The correlation of the laboratory stability test with engine performance is sufficiently satisfactory to justify further work. In this connection, an automatic temperature control is being provided so that the test may be run overnight and hence reduce the time of test in terms of work days.

Sampling Plan Reduces Inspection Costs

Whenever industrial and agricultural products are purchased in quantity, the buyer must employ some sort of inspection procedure to determine whether or not the material meets the specified standard of quality. The extent of this inspection will depend on the nature of the material and may range all the way from a rough appraisal based on an examination of a few items, as in the case of chinaware in wholesale lots, to the complete inspection of each item, when buying industrial diamonds, for example. The decision to accept or reject a given lot of material is often determined by the average quality of a random sample taken from the lot. Wherever such a routine acceptance test is conducted, any way of reducing the amount of inspection, and therefore the cost, is worth exploring.

A modified double sampling plan has been developed by J. M. Cameron and W. J. Youden in the Bureau's Statistical Engineering Laboratory that makes possible a saving in the average amount of inspection that may range from 20 to 40 percent. In this new scheme, the regular testing procedure is interrupted after a predetermined number of units have been examined. The material is accepted if the test results at the time of interruption fulfill the criteria of a simple rule that requires neither computation nor the use of tables. If the material does not pass this test, the inspection is completed in the usual manner. In either case, the inspection procedure itself remains unchanged.

One common inspection method involves "single sampling": drawing a single sample of a fixed number of units at random from the material under test. Acceptance or rejection may depend upon whether or not the average of the individual test results is greater than a predetermined limit, or upon whether the number of "good" items exceeds a fixed acceptance number. The first method is usually referred to as "variables inspection," the second as "attributes inspection." In either case, only a single acceptance number is needed.

Whether inspection is based on "variables" or "attributes," a reduction in the average amount of inspec-

tion can be achieved by interrupting the single sampling process at some point before completion and weighing the evidence accumulated up to that point. In the case of ordinary "double sampling" this interruption is made at a predetermined point, and if the test results are sufficiently one-sided, the material is either accepted or rejected. If the evidence is indecisive, a fixed number of additional units is drawn, and the results are combined and treated as in single sampling.

Another inspection scheme in common use is "sequential sampling." In sequential sampling the test results are accumulated unit by unit until the evidence is sufficiently one-sided to justify acceptance or rejection of the lot. A table or graph of acceptance and rejection values for sequential sampling is needed at each stage of the testing procedure, whereas ordinary double sampling requires acceptance and rejection values for the first sample, and the acceptance value for the combined first and second samples.

Replacement of the single sample plan by an ordinary double or sequential plan involves a change in the number of units to be drawn from the material under inspection. In addition, for "variables inspection" the double and sequential plans require the use of tables or more computation in order to arrive at a decision to accept or reject.

Features of the New Plan

The modified double sampling plan developed at this Bureau is a substitute for the usual single sampling plan and does not alter the sampling itself or require additional computation. In this new plan the same number of units is drawn as in the single sample case, but the regular testing procedure is interrupted after a fixed number of items have been tested. If a predetermined number of these test results individually exceed the acceptance value of the original plan, the material is immediately accepted and testing stopped. If less than the required number meet the acceptance value, testing is completed in the usual way and the individual test results are averaged. Acceptance or rejection then depends on whether or not this average exceeds the acceptance value.

For example, in the acceptance testing of transformer oil it might be required that the *average* breakdown voltage exceed 25 kilovolts for five cups of oil drawn at random from a large container. Under the modified double sampling plan (with the assumption that the allowable risk of accepting unserviceable oil is 0.05), if the first two cups *both* exceed 25 kilovolts in breakdown strength the lot is accepted. If either of these first two samples break down below 25 kilovolts, three more cups are tested and the average of the five breakdown values must exceed 25 kilovolts for acceptance of the lot. Whenever oil is accepted on the basis of two cups alone there is a saving of 60 percent in the amount of inspection.

The chance that material of a given quality will be accepted under the modified double sampling plan is thus slightly larger than on the original single sample plan, since there will be some samples that have the required number of items exceeding the acceptance value at the point where inspection is interrupted, but which, if completely inspected, would have yielded an over-all average below the acceptance value. This increase in the chance of acceptance is desirable when the general quality of the material under inspection is known to be "good." It is undesirable when the quality is "bad," and this slight increase in the chance of accepting bad material is the price to be paid for reducing the average amount of inspection without disturbing existing routines.

Typical Inspection Savings

A properly designed single sampling plan is usually set up so that the chance of accepting unserviceable material is suitably small, say, 0.01 or 0.05. No sampling plan can guarantee the rejection of unserviceable material, but the chance of accepting such material can be made as small as desired by properly choosing the acceptance value and the number of units to be tested. The table gives the Bureau's modified double sampling plan to replace the single sample plan when the allowance risk of accepting bad material is either 0.01 or 0.05. The last column shows the average percentage saving in the number of units tested when the material is of sufficiently good quality to be accepted about 95 percent of the time under the original inspection procedure. In this table, if the original single sample plan calls for a number between the tabulated values of n, the next higher value is to be used.

r	n	m	k	Saving
				%
0.01	4	2	2	22
	9	3	3	24
	10	5	4	26
	11	7	5	23
	19	6	5	24
.05	5	2	2	35
	6	4	3	25
	8	6	4	20
	17	5	4	31
	24	3	3	23
	43	8	6	25

r = allowable risk of accepting unserviceable material.

n = number of items drawn in original single sample plan.

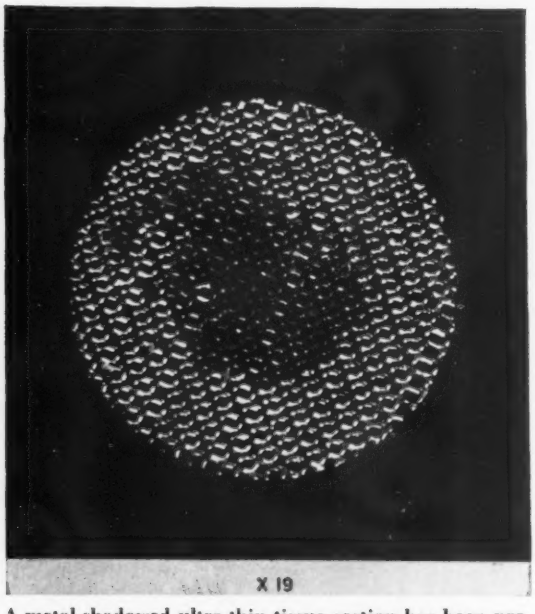
m = number of items to be examined in modified plan before interrupting tests.

k = number of items in m that must individually satisfy the acceptance value if the lot is to be accepted at this point. If less than k items qualify, testing should be completed as in original plan and the material accepted if the average of the n values exceeds the acceptance value.

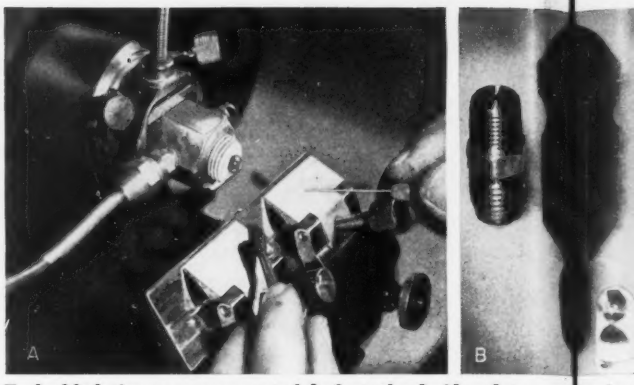
A new method in ultra-microtomy, developed by S. B. Newman, Emil Borysko, and Max Swerdlow of the National Bureau of Standards, now provides an inexpensive, practical means of preparing very thin sections of biological tissue for study with either the light or electron microscope. In the new procedure, *n*-butyl methacrylate is polymerized around the specimen to produce an optically clear embedding medium having highly desirable cutting properties. A smooth, continuous advance of the specimen toward the knife of a slightly modified conventional microtome is then obtained from the thermal expansion of a metal specimen holder. Thus, sections of tissue having undistorted structure and a uniform thickness of fractions of a micron over a large area may be cut one at a time.

To medical and biological investigators, who use the electron microscope in such fields as cancer research and the study of virus diseases, the need of an efficient method of producing uniform thin sections of tissue has been apparent for several years. Indeed, the application of the electron microscope to many biological problems has been seriously hampered by the lack of such a method. Because of the very slight penetrating power of the electron beam in commercial instruments and the great relative depth of field involved, specimen structure is difficult to interpret when sections are over a fraction of a micron in thickness. Yet the techniques that have been available for preparing thin sections are quite elaborate and difficult, requiring expensive equipment and producing few usable sections.

Of the various solutions to this problem that have been suggested, each has apparently been applicable only to a selected group of materials. One rather



A metal-shadowed ultra-thin tissue section has been prepared for electron microscopy by centering on a ⅛-inch 200-mesh specimen screen.



Embedded tissues are mounted before the knife of a conve micro Bureau for cutting extremely thin sections (A). The speci lder i enters from the tube at left. Thermal expansion of the holde advanc sembled view of the specimen holder: Brass block (upper ce the ⅜ valve for the carbon dioxide gas (left and right), and the emb specin dry over the thin sections on a glass slide and is then floated slide o have been placed over the areas of the film containing the se

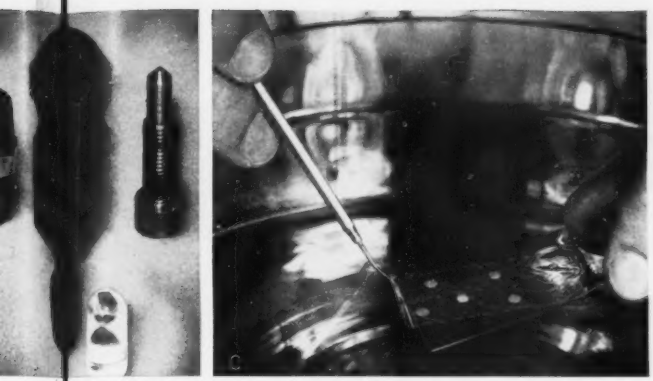
NBS Method f Mi

successful approach was the high-speed microtome, in which the cutting edge revolves at tremendous speed; but this equipment, besides being expensive and complicated, produces a low percentage of good sections. Moreover, the sections are cut so rapidly and abundantly that selection is tedious and uncertain. Several workers have cut wedge-shaped sections, using the thinnest portions for electron microscopy. Their methods, however, have been laborious and difficult to reproduce.

The most recent effort has been that of Pease and Baker,[2] who used a modified Spencer rotary microtome. These investigators reduced the unit of specimen advance to approximately one-tenth of the calibrated value. The microtome was then reported to produce sections as thin as 0.1 micron from tissue embedded in paraffin and collodion. Many workers, however, have found it difficult to use this technique, mainly because of the exacting demands made on the microtome-advancing mechanism. Another disadvantage has been the difficulty of making very thin sections with the standard embedding media, such as paraffin and collodion. The Bureau therefore initiated the development of a rapid, efficient sectioning procedure that could be carried out in routine fashion by an operator without a great deal of special training. The resulting method, which goes far in accomplishing this objective, should be of decided advantage in those fields of biology, medicine, agriculture, and industrial technology that are concerned with the microstructure of plant and animal material.

An important feature of the Bureau's method is an easily made, inexpensive device that holds the embedded specimen and at the same time advances it gradually and uniformly toward the knife of the micro-

[2] D. C. Pease and R. F. Baker, Proc. Soc. Exp. Biol. Med. **67,** 470 (1948).



a conve microtome in the specimen-holding device developed at the e speci older is cooled by the expansion of carbon dioxide gas, which e holde dvances the specimen smoothly and continuously. (B) Disas- pper ce the ⅜-inch pipe plug (lower center), the two parts of the needle the emb specimen (right center). (C) A collodion film is allowed to floated slide onto water. The small round electron microscope screens the sec

d f Microsectioning

tome. This device may be employed without modifying the conventional microtome other than to disengage the mechanism normally used to advance the specimen. It is essentially a brass block containing a hole threaded at one end to receive a standard ⅜-inch brass pipe plug. A cavity drilled into the face of the plug provides a seat for the embedded specimen. Behind the plug is a needle valve, which admits compressed carbon dioxide gas to the interior of the block. As the gas undergoes a large increase in volume, it cools and contracts the assembly. Stopping or reducing the flow of carbon dioxide allows the apparatus to approach room temperature, and thus the thermal expansion provides continuous advance of the embedded tissue toward the cutting edge.

In the procedure developed at the Bureau, the tissues are fixed and then dehydrated in an ethyl alcohol series by the usual cytological techniques. From absolute alcohol they are transferred to a solution containing equal parts of absolute alcohol and monomeric *n*-butyl methacrylate. After about 1 hour in the alcohol-monomer mixture, the tissue is put in the monomer alone for an equal period. To ensure removal of the alcohol they are then placed in two additional changes of monomer for at least 1 hour each before embedding in the polymer.

Gelatin capsules serve as convenient embedding molds. The main body of the capsule is set upright in a wooden block or other base and filled with the monomer, to which has been added 1 percent (by weight) of a polymerization catalyst (2,4-dichlorobenzoyl peroxide). After the tissue is placed in the mixture, the lid of the capsule is slipped on to retard evaporation. The assembled capsules are then suspended by strips of cellophane tape in an oven kept at a temperature of 45° to 50° C.

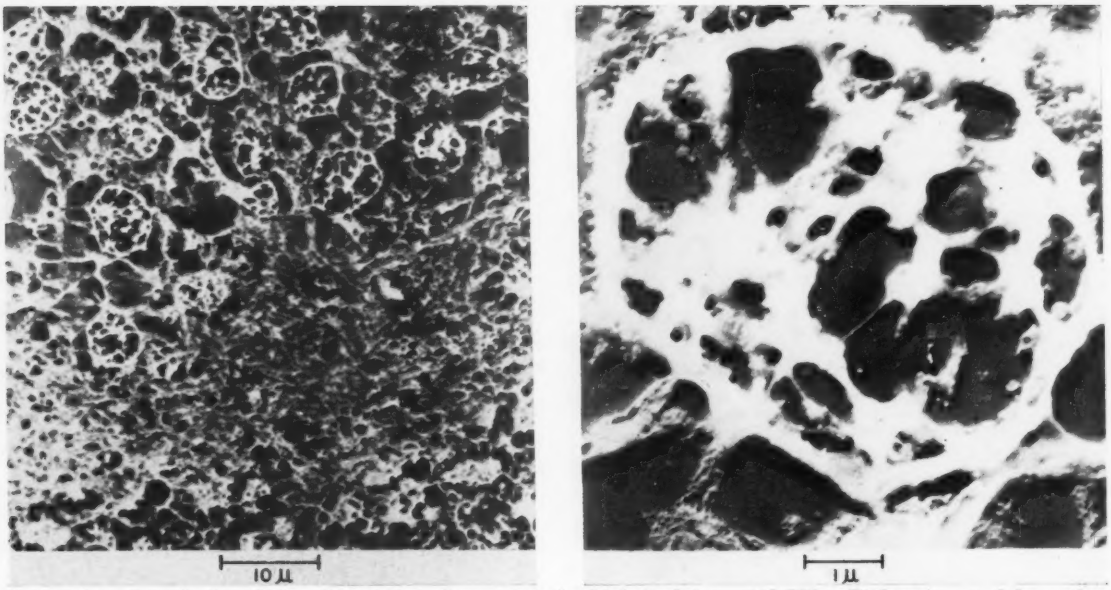
After 6 or 8 hours of heating, the monomer is polymerized into a solid matrix containing the tissue embedded at the bottom of the clear plastic. Several hours more at this temperature will ensure complete cure. The gelatin capsule may then be removed by soaking in water.

The embedded specimen is cemented into the mounting block with a mixture of pure gum rubber and paraffin. Then, with the thermal-expansion device clamped in the jaws of the microtome head, the entire assembly is cooled below room temperature. Upon the appearance of a thin layer of frost on the metal, the knife is adjusted so that the specimen just misses it on the cutting stroke. The specimen is then mechanically advanced at 2- or 3-micron increments until the first slice is made. At this point the mechanical advancing mechanism of the microtome is disengaged by setting it to zero and the gas flow reduced or stopped. The specimen then advances because of the thermal expansion of the metal holder. With a little experience the operator can soon judge the necessary time interval between cuts. Some control of the rate of specimen advance can be obtained by bleeding the carbon dioxide at various reduced rates into the expansion chamber.

Although polybutyl methacrylate has excellent cutting properties, the cut sections usually are found to be somewhat folded. They are lifted from the knife by a dry camel's hair brush, picked up with a dissecting needle, and placed on a water surface warmed on a hot plate to about 35° C. After a period ranging from a few minutes to an hour or more, many will flatten out on the surface. Those sections that are thin enough to show bright interference colors are then floated onto clean microscope slides and allowed to dry flat.

Sections prepared for phase-contrast microscopy are placed in acetone or toluene for about ½ hour to remove the matrix and then mounted in Canada balsam. For

Detection of structural details in some specimens is greatly improved by metallic shadow-casting. Chromium is evaporated from the electrically heated basket (center) under high vacuum and condenses, forming radiation shadows, on the tissue sections that rest upon specimen screens (left, on strips of paper).



Left: Chromium-shadowed ultra-thin section of representative biological tissue, ×1,500. Right: A part of the section at left showing a cell nucleus, magnified 10,400 times.

ordinary light microscopy the matrix is dissolved and the section stained in the usual way.

In preparing material for the electron microscope, the sections are floated from the water onto clean glass slides and dried flat. The matrix is then dissolved by placing the slide in acetone, toluene, or amyl acetate. A dilute solution of collodion in amyl acetate is allowed to flow over the slide bearing the tissue, which is then permitted to dry at room temperature. The collodion film is floated from the slide onto water, and electron-microscope specimen screens are placed over the areas of the film containing the sections. The entire film is then floated from the plate to a larger volume of water, and the freely floating film, to which the specimen screens adhere, is removed from the water by flipping it over and out with a strip of short-fibered paper or a glass microscope slide.

The detection of structural details in some of the biological tissues sectioned at the Bureau was greatly improved by metallic shadow-casting in the manner of Williams and Wyckoff. This process, involving the angular deposition of a thin metallic layer on the section, produces a three-dimensional aspect as well as greater contrast in the structural details of the tissues. The sections are freed of the embedding media and stripped from the glass slide with a collodion film as described previously. They are then mounted, tissue side up, on the specimen screens and exposed to a metallic vapor under high vacuum. Material prepared for light microscopy may be stained and shadowed before mounting.

Although the Bureau's new method for obtaining very thin sections has given excellent results, it possesses certain limitations. The greatest chance for failure appears to lie in the polymerization of the embedding mass. However, use of low-temperature catalysts and maintenance of a curing temperature of 45° to 50° C will minimize difficulties at this stage. Occasionally tissues are injured during the polymerization reaction, but such tissues, which are easily detectable, can be promptly discarded. The tilt of the knife during sectioning is not particularly critical. Obviously the knife must be smooth, clean, and freshly sharpened. Satisfactory edges are maintained by commercial sharpeners. On the whole, while fixation artifacts still remain problems for serious consideration, the new method makes possible the production of a high percentage of usable sections that have uniform thickness over a large area and integrity of tissue structure. Efforts are now being made at the Bureau to apply the new sectioning method to the study of the structure of natural and artificial fibers found in leather, paper, and textiles.

Unified Screw Thread Standards

The proceedings of the Joint Session of the Representatives of Canada, United Kingdom, and United States on the unification of screw threads have been published by the National Bureau of Standards. International trade in equipment of all kinds has been hindered by the lack of interchangeability of screw thread parts. Adoption of these standards will remove an important barrier to the exchange of manufactured goods throughout the world.

Circular 479, *Unified Screw Thread Standards*, 27 pages, illustrated with tables and figures, is available from the Superintendent of Documents, Government Printing Office, Washington 25, D. C., at 30 cents a copy. Foreign orders must include one-third the price for postage.

Effect of Boron on the Structure and Properties of Cast Iron

War-induced scarcities of other elements called attention to the possible use of boron for increasing the hardness and wear resistance of cast iron. However, specific data regarding the effect of boron on the structure and properties of cast irons have been somewhat limited. Recently A. I. Krynitsky and Harry Stern of the National Bureau's experimental foundry made an extensive study [3] of the effect of boron in plain gray cast irons, obtaining useful information on solidification characteristics, strength, hardness, microstructure, and chilling properties. These data should be of value to those industries that produce cast iron for specialized uses requiring definite characteristics.

Cast iron consists essentially of graphite particles dispersed throughout a steel matrix. Ordinarily the fractured surface of the metal is gray, giving rise to the term "gray iron"; but when cast iron is cooled rapidly from the molten state, it "chills", forming "white iron." Because of its hardness and resistance to wear, white cast iron is used in such articles as street-car wheels and rolls for rolling mills. The core of these castings consists of soft gray iron, while the outside portion is the more durable white iron.

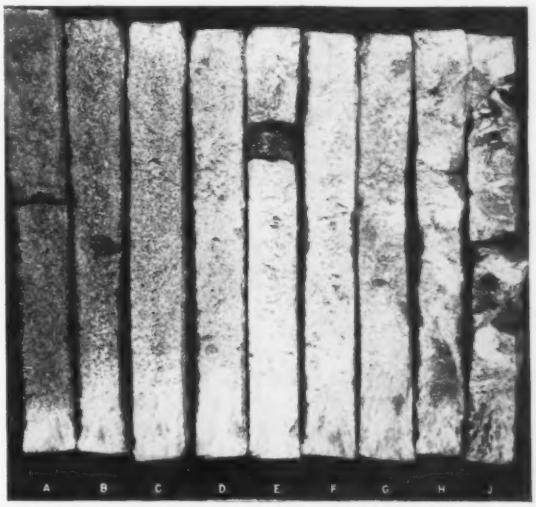
The brittleness of the iron chip and the lubricating properties of the graphite make gray iron easy to machine. The graphite particles also act as cushions against thermal shock and thus prevent crazing. For this reason, gray iron is the only material that has been successfully used on a large scale for brake shoes or brake drum linings, which must absorb tremendous energy, convert it to heat, and still not seize the wheel or axle.

In the Bureau's investigation, plain gray cast irons of nine different stocks were studied. The carbon content in these irons varied from 3.3 to 4.1 percent, and the silicon content from 1.4 to 2.7 percent. The irons were melted in high-frequency induction furnaces, and boron in the form of ferro-boron was added to the molten metal in amounts ranging from less than 0.001 percent up to 0.48 percent. Green, dry, and core sands were used in preparing the molds.

The solidification characteristics of a number of heats were determined by making "inverse rate" cooling curves under as nearly uniform conditions as possible. The curves indicated that undercooling increases with an increase in boron. At higher percentages of boron, the composition seems to approach a eutectic point, where the liquidus and solidus are merged.

When examined under a microscope, the structure of irons with a relatively large boron content was found to consist essentially of a matrix of cementite (Fe_3C) containing islands of pearlite (a mixture of ferrite and Fe_3C) as well as other structureless islands. The pearlite areas were usually surrounded by a structureless band. In order to ascertain the nature of these structureless islands and envelopes, a series of experiments, consisting of a liquid air quench, an annealing treatment, and magnetic studies, was carried out. It seems probable that the envelopes surrounding the pearlite areas are ferrite, while the structureless islands are very fine pearlite that could not be resolved by the microscope. It is also probable that the various constituents—pearlite, ferrite, and cementite—are of a complex nature, containing boron in solid solution or in the form of a compound.

Transverse strength was determined by subjecting bars 1.2 inches in diameter to a breaking test. A disk $\frac{3}{4}$ inch thick was then cut from each broken transverse test bar adjacent to the fracture and used for Brinell hardness determinations and for metallographic examination. Fractures could be classified as gray, white-network, or mottled. For bars with gray fractures, an increase in boron content was accompanied by a small increase in the transverse strength, Brinell hardness, and relative modulus of elasticity of the sample. However, the appearance of a white network or white specks on the fractured surfaces of bars containing larger amounts of boron usually was accompanied by a



The fractured surfaces of experimental chill plates cast with their lower edge against an iron chill block show the effect of gradually increasing amounts of boron in cast iron. The whiteness of the castings at right, which contain the most boron, indicates that these specimens have greater ability to chill to the harder, more durable, white iron. The boron contents vary from 0.002 percent (A) to 0.48 percent (J).

[3] For further technical details, see Effect of boron on the structure and some physical properties of plain cast irons, by Alexander I. Krynitsky and Harry Stern, J. Research NBS **42**, 465 (1949) RP1987.

drop in the transverse strength and deflection and an increase in the Brinell hardness and relative modulus of elasticity. Brinell numbers varied from 140 to 220 for bars with gray fractures, from 220 to 285 for bars having a white network, and from 295 to 427 for those with mottled and almost white fractures.

Another very important property of cast iron is its ability to chill with the formation of white iron. Depth of chill may be controlled in two ways—by varying the cooling rate or by adjusting the composition of the iron. In the present study, the whiteness of the fractured surface of plate and wedge specimens was taken as a criterion of the iron's susceptibility to chill. Plates 4×4 inches square and ½ inch thick were cast with one edge against a heavy iron chill block and were broken at the middle, perpendicular to the chilled side, to measure the depth of chill. Wedge specimens 2½ inches wide at the base, 3 inches high, and 4 inches long, cast in dry sand molds, were also broken at the middle. For all castings made under similar melting and pouring conditions, there was a pronounced increase in depth of chill with an increase in the boron content. This was found to be accompanied by a greater development of the white network in the gray portions of the castings. The chilled areas of the plates usually consisted of two zones: a completely white zone, known as "clear chill", and a partly chilled or mottled zone. For irons containing a large amount of boron—0.32 percent and higher—the chill plates proved inadequate for measuring the depth of chill. Fractures of plates containing 0.32 and 0.35 percent of boron were mottled white while those containing 0.37 and 0.48 percent of boron were wholly white. In the wedges containing 0.35, 0.37, and 0.48 percent of boron the mottled zones were absent, and the white portion of the wedge containing 0.48 percent of boron consisted of a massive columnar structure.

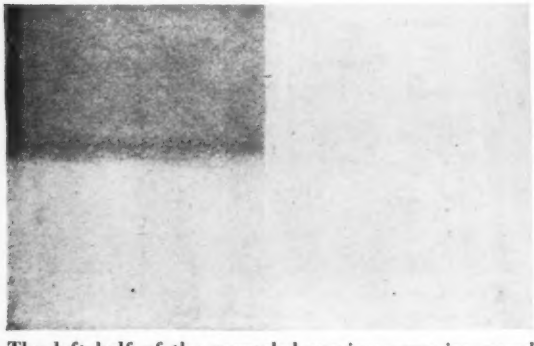
Vickers hardness determinations made on the white portions of the chill-plate specimens showed an increase in Vickers number with an increase in boron content. For the chill plate containing 0.48 percent of boron the Vickers hardness number was 721—corresponding to a number between 620 and 650 on the Brinell scale—while for regular white iron the Brinell hardness number lies between 320 and 530.

In general, the results of the investigation show that boron increases the depth of chill of cast iron as well as its hardness. Boron was also found to decrease the size and quantity of graphite particles, to increase the free carbide content, and to produce an undercooling effect that becomes more pronounced with greater percentages of boron.

Effects of Acid Treatment on Abrasion and Acid Resistance of Porcelain Enamels

Recent work by W. N. Harrison, J. C. Richmond, and J. R. Crandall of the Bureau's Enameled Metals Laboratory, has revealed interesting effects of acid pretreatment of porcelain enamels on the acid resistance and abrasion resistance of the treated specimens. While full explanations are yet to be determined, current interest in these phenomena warrants publication of the results so far obtained.

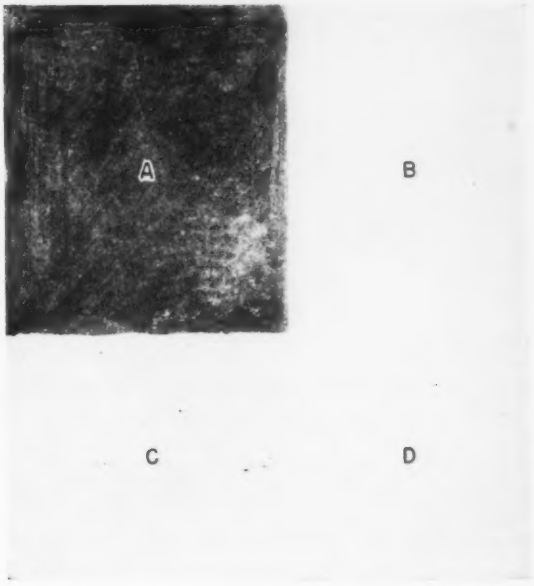
In the revision of the Federal Specification for enameled graduates for darkroom use, tests were made to compare the relative effects of hydrochloric, acetic, and citric acids on a number of enamels. These tests indicated that acetic acid was much less corrosive than were hydrochloric and citric acids. The most significant finding, however, was that treatment with acetic acid, which produced only minor visible attack, strongly inhibited further attack when subsequently treated with citric acid, although the citric acid severely attacked the untreated areas of the same enameled specimens. It was found that pretreatment with acetic acid, in concentrations from 0.5 to 50 percent for periods of 5 minutes or longer, was effective against subsequent treatment with 10-percent citric acid. Treatment with butyric acid for 15 minutes produced the same effect, but neither tartaric nor lactic acid was effective.



The left half of the enameled specimen was immersed in 10-percent citric acid for 15 minutes, after which it was abraded by hand across its entire width. The specimen was marked with a No. 1 lead pencil over its entire surface to accentuate the abraded and unabraded areas.

This passivation effect may be explained on the hypothesis that the acetic and butyric acids preferentially leach alkalies from the enamel surface, leaving behind a silica-rich film that is resistant to further solution even in citric or stronger acids. With citric acid, on the other hand, the attack apparently proceeds to a greater depth before sufficient thickness of this film is built up to inhibit further solution.

It was found that treatment for 15 minutes in 10-percent citric acid appreciably reduced the abrasion resistance of certain enamels having Class B or Class A acid resistance (Porcelain Enamel Institute standard



An enameled specimen shows the effects of treatment with 10-percent acetic acid, followed by treatment with 10-percent citric acid. (A) treated with citric acid only; (B) untreated; (C) treated with acetic acid followed by citric acid; (D) treated with acetic acid only. Note the protection afforded by pretreatment with acetic acid (C). Specimen was marked with a No. 1 lead pencil to accentuate the etching.

test), this effect being more pronounced on titanium-type enamels than on antimony-type enamels. In no case was the abrasion resistance of Class AA enamels affected.

The effect of abrasion (such as would occur, for instance, when a stained spot on an enameled article is scoured) was sufficient to reduce the rating on some enamels from a good Class B by the standard test to a poor Class C by the same test, modified to include the abrasion treatment. The same amount of abrasion had no appreciable effect on the untreated areas of the specimens.

It is believed that these two effects are related, and that the silica-rich layer resulting from preferential solution of the alkali is less resistant to abrasion than the original surface, although more resistant to acid attack.

The results suggest that titanium-type enamels are especially sensitive to the observed effects, and that routine inspection tests should be made in regular production to assure that the composition and processing of the enamels are maintained at optimum conditions.

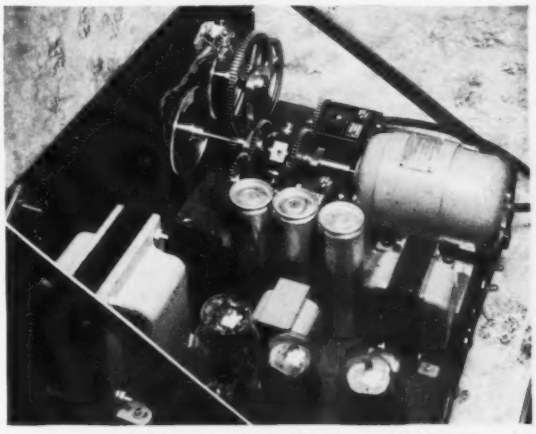
The increased use of titanium-type enamels and the sensitivity of these enamels to attack by abrasion after treatment with acid indicate that an abrasion treatment should be incorporated in the standard test for acid resistance.

Frequency Monitoring Device for Interval Timers

A frequency monitoring instrument developed by R. E. Gould and H. A. Bowman of the Bureau's Time Section now provides a means by which any Bureau laboratory may obtain the proper correction to be applied to a commercial interval timer driven by the power line. Operation of the device is simple and straightforward, offering a minimum of opportunity for error in the correction given. Since an accuracy of 0.01 second is thus obtained, the new development has extended the usefulness of interval timers at the Bureau to the realm of precise measurements.

Interval timers driven by synchronous motors connected to the power line are widely used in science and technology for timing radiation counters, fluid flow in hydraulics, viscosity measurements, chemical reactions, ballistics experiments, and high-speed photography. Yet one serious disadvantage in their use has been the error caused by variations in the frequency of the current in the local power system. The new frequency error indicator has eliminated this difficulty at the Bureau; similar devices should prove of decided advantage in other laboratories where accurate timing of experiments is important.

The Bureau's design makes use of the difference in speed of rotation of two 60-cycle synchronous motors, one driven by the power line and the other by a standard crystal-controlled frequency. The two motors are connected to a differential gear system. The angular velocity of the differential gear shaft thus depends on the departure of the power line frequency from the standard frequency. Two standard frequencies would



A frequency-monitoring device has been developed at the Bureau for accurate correction of the integral timers used in its laboratories. Any difference in the speeds of the two small black synchronous motors results in motion of the differential gear shaft between them. This shaft is connected to plastic indicating dials in the face of the instrument.

hold the gear shaft motionless. The position of this gear shaft at any instant depends upon the total integrated frequency error of the power line at that instant. This shaft is connected to indicating dials, which are calibrated to give the cumulative error in seconds and hundredths of a second rather than in cycles.

In practice, the individual at the Bureau who wishes to time an experiment may call the Bureau's time laboratory both at the beginning and end of the experiment to obtain the correction given on the dials of the error indicator. The initial and final corrections, each a positive number, are then added to the initial and terminal observations, respectively, of the interval timer that is used. The difference between these two sums is the actual duration of the interval measured. Since the cumulative error seldom drifts faster than 0.30 second per minute, the correction received by any Bureau laboratory in this way is precise within a very few thousandths of a second. An automatic recording device operated in conjunction with the system provides a complete charting of the frequency variation over the time interval when desired.

NBS Publications

Periodicals [4]

Journal of Research of the National Bureau of Standards, volume 43, number 1, July 1949. (RP2000 to RP2009, inclusive).

Technical News Bulletin, volume 33, number 7, July 1949. 10 cents.

CRPL–D59. Basic Radio Propagation Predictions for October 1949. Three months in advance. Issued July 1949, 10 cents.

Nonperiodical [4]

Research Papers [4,5]

RP1991. A study of laboratory Bunsen burners for natural gas. John H. Eiseman. 10 cents.

RP1992. Studies on the purification of collagen. James M. Cassel and Joseph R. Kanagy. 10 cents.

RP1993. Flame spectrum of acetylene from 1 to 5 microns. Earle K. Plyler and Curtis J. Humphreys. 10 cents.

RP1994. Viscosity of deuterium oxide and water in the range 5° to 125° C. Robert C. Hardy and Robert L. Cottington. 10 cents.

RP1995. Determination of efficiency of microwave bolometer mounts from impedance data. David M. Kerns. 10 cents.

RP1996. Ultraviolet absorption spectra of seven substituted benzenes. Ralph Stair. 10 cents.

RP1997. Acid-base equilibrium constants for the reaction of tribenzylamine with picric acid and with trinitro-*m*-cresol in benzene, from spectrophotometric data. Marion Maclean Davis and E. Anne McDonald. 10 cents.

RP1998. Transmission of reverberant sound through single walls. Albert London. 10 cents.

RP1999. Separation and identification of the major C_7 to C_{10} components of triptene residue. Philip Pomerantz, Thomas W. Mears, and Frank L. Howard. 10 cents.

Commercial Standards [4]

CS154E–49. Wire rope (Export classification). 15 cents.

CS158–49. Model forms for girls' apparel. 10 cents.

CS159–49. Sun glass lenses made of ground and polished plate glass thereafter thermally cured. 10 cents.

Simplified Practice Recommendations [4]

R155–49. Cans for fruits and vegetables. (Names, dimensions, capacities, and designated use). (Supersedes R155–40). 10 cents.

R207–49. Pipes, ducts, and fittings for warm-air heating and air conditioning. (Supersedes R207–45). 10 cents.

R236–49. Delivery cases for square glass milk bottles. 10 cents.

Circular [4]

C479. Unified screw thread standards. 20 cents.

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Natural and synthetic rubbers. Norman Bekkedahl and Robert D. Stiehler. Analytical Chemistry (1155 Sixteenth Street, NW., Washington 6, D. C.) **21,** 266 (1949).

Some problems in the precise measurement of pressure. W. G. Brombacher. Instruments (1117 Wolfendale Street, Pittsburgh 12, Pa.) **22,** 358 (1949).

An easily assembled console for rapid testing of electrical indicating instruments. Frank D. Weaver. Instruments, **22,** 396 (1949).

Note on the preparation and testing of hydrocarbon solvents for absorption spectroscopy. Marion Maclean Davis. The Analyst (Cambridge, England) **73,** 395 (1948).

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Deterioration of cotton textiles exposed to the weather. Herman Bogaty. Am. Dyestuff Reporter (1 Madison Ave., New York 10, N. Y.) **38,** 253 (1949).

Reduction of sunspot-number observations. A. H. Shapley. Publications of the Astronomical Society of the Pacific (Palo Alto, Calif.) **61,** No. 358, 13 (February 1949).

The spectral-transmissive characteristics of some German glasses. R. Stair, F. W. Glaze, and J. J. Ball. The Glass Industry (55 West Forty-second Street, New York 18, N. Y.) **30,** 331 (1949).

Meson mass and range of nuclear forces. U. Fano. Am. J. Phys. (57 East Fifty-fifth Street, New York 22, N. Y.) **17,** 318 (1949).

Flammability varies with type test and fabric. Marjorie W. Sandholzer. Textile World (330 West Forty-second Street, New York 18, N. Y.) **98,** No. 3, 109 (1948).

Lightweight-aggregate concrete. R. W. Kluge, M. M. Sparks, and E. C. Tuma. J. Am. Concrete Inst. (7400 Second Boulevard, Detroit 2, Mich.) **20,** 625 (1949). Title No. 45–37.

Theory of imperfect diffraction gratings. U. Fano. J. Opt. Soc. Am. (57 East Fifty-fifth Street, New York 22, N. Y.) **38,** 921 (1948).

Absorption by sound-absorbent spheres. R. K. Cook and Peter Chrzanowski. J. Acoustical Soc. Am. (57 East Fifty-fifth Street, New York 22, N. Y.) **21,** 167 (1949).

[4] Send orders for publications under this heading only to the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Annual subscription rates: Journal of Research, $4.50 (foreign $5.50); Technical News Bulletin, $1.00 (foreign $1.35); Basic Radio Propagation Predictions, $1.00 (foreign $1.25). Single copy prices of publications are indicated in the lists.

[5] Reprints from June Journal of Research.

[6] These publications are not available from the Government. Requests should be sent direct to the publishers.

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